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ENVIRONMENTAL SERVICES

Supplemental Mercury Methylation and Remineralization Studies

Work Plan

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SUPPLEMENTAL MERCURY METHYLATION AND REMINERALIZATION STUDIES

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INTRODUCTION

During a meeting with the New York State Department of Environmental Conservation (NYSDEC), the U.S. Environmental Protection Agency (EPA), AlliedSignal Inc (AlliedSignal), PTI Environmental Services (PTI), and external reviewers held on October 12, 1995, it was agreed that the collection of a limited amount of supplemental data should provide the basis for completion of the mercury model for Onondaga Lake. This work plan therefore proposes additional determinations of water column methylation rates and remineralization rates in Onondaga Lake. Confirmation of previous estimates of these rates will allow finalization of the Onondaga Lake Mercury Model, as discussed in the October 12, 1995, meeting (Bigham 1995a, pers. Comm.). This work plan presents the rationale and experimental design for the 1996 studies of mercury methylation and remineralization in Onondaga Lake. These studies, conducted on behalf of AlliedSignal as part of the Onondaga Lake remedial investigation and feasibility study (RI/FS), represent a refinement of the experimental work performed for the RI/FS in 1992.

The results of field work and experiments conducted in 1992 for the Onondaga Lake RI/FS indicate that 1) net methylmercury production in the water column is the major source (i.e., 69 percent) of methylmercury to the lake, 2) dissolved flux of total mercury and methylmercury to the lake from sediments is a minor (i.e., less than 1 and 2 percent of total, respectively) source to the lake, and 3) remineralization, as determined from comparison of gross and net sedimentation, is a significant source of methylmercury to the hypolimnion (Henry et al. 1995; PTI 1994). The field work and experiments conducted in 1992 used "state-of-the-art" methods; nevertheless, data from these experiments were variable and techniques have been refined since that time. Thus, it is useful to confirm the earlier results.

SAMPLING OBJECTIVES AND EXPERIMENTAL APPROACH

Onondaga Lake, like other stratified lakes, experiences a buildup of total mercury and methylmercury in the hypolimnion during summer stratification (Bloom et al. 1991; Cossa et al. 1994; Verta and Matilainen 1995; Verta et al. 1994; Watras and Bloom 1994; Watras et al. 1995). This buildup can result from the following processes:

- Methylmercury production in the water column
- Transport of mercury from the epilimnion on settling particles (followed by remineralization or release of mercury from these settling particles)
- Direct input of mercury from tributary loading to the hypolimnion
- Flux of dissolved mercury from sediments
- Resuspension of sediments containing mercury
- Advective flux of mercury from groundwater.

The objective of the proposed 1996 field work is to better define net water column methylmercury production rates and rates of remineralization of total mercury and methylmercury in the hypolimnion. These processes contribute significantly to the buildup of total mercury and methylmercury in the hypolimnion of Onondaga Lake based on analysis of the 1992 data (Henry et al. 1995; PTI 1994). Direct tributary loading to the hypolimnion is currently being calculated based on density profiles. Conducting additional experimental work for estimating dissolved flux was deemed to be of little benefit, as discussed later in this section. Resuspension is considered to be insignificant in the profundal zone because waters below 6–8 m are quiescent and an area of net deposition (Auer et al. 1996). Advective flux from groundwater is also considered to be negligible based on estimates of groundwater loading discussed in *Onondaga Lake RI/FS Mercury Modeling Report* (PTI 1994).

Net Methylmercury Production in the Water Column

Estimation of net methylmercury production in the water column is generally accomplished by measuring the increase in methylmercury concentration in water samples over time. Three refinements of the 1992 experiments for net methylmercury production in the water column (PTI 1992) will be included in 1996. First, containers of lake water will be incubated in the field rather than in the laboratory. This approach has been used successfully in other lakes for measurement of mercury methylation (Matilainen 1995; Watras et al. 1995). The advantages of *in situ* incubation over laboratory incubation include better simulation of field conditions and reduction in the amount of sample manipulation.

Second, rather than the ambient approach where methylmercury is produced from mercury already present in the sample (as was done in 1992; Watras et al. 1995), a radioisotope of mercury (^{203}Hg) will be added to sample containers at background concentrations. Whole-water samples will then be analyzed for ^{203}Hg-methylmercury over time. High specific activity ^{203}Hg is now available and has been used for estimating methylation rates in water and sediment samples (Gilmour and Riedel 1995; Stordal and Gill 1995).

Previously, the low specific activity of commercially available ^{203}Hg limited interpretation of methylation experiments because it was necessary to add mercury concentrations that were much greater than background (Gilmour and Henry 1991). The radioisotope approach greatly simplifies methylmercury analysis.

Third, studies since 1992 have indicated that most of the increase in methylmercury occurs within 24 hours of incubation (Gilmour and Riedel 1995). Therefore, samples will be incubated for a total of 48 hours with more intensive subsampling during the first 24 hours. The combination of *in situ* incubation age of ^{203}Hg with high specific activity and intensive subsampling within 24 hours should yield reliable estimates of net methylmercury production in the water column.

Remineralization

Remineralization in this work plan refers to the release of total mercury and methylmercury from settling particles in the hypolimnion. The process has also been termed recycling and has been described for elements other than mercury. Remineralization is well-documented for phosphorus in the water column of lakes. For example, Wodka et al. (1985) determined that phosphorus settling from the epilimnion to the hypolimnion in Onondaga Lake cycles about three times before it is incorporated into sediment or exported from the lake. The authors refer to high dissolved phosphorus concentrations in the hypolimnion during stratification as support for vertical cycling of phosphorus in Onondaga Lake. Similarly, the increase in dissolved total mercury and methylmercury concentrations in the hypolimnion during summer stratification may reflect mercury remineralization/recycling processes (Hurley et al. 1991; Hurley et al. 1994; Henry et al. 1995). The exact location of the remineralization process in the hypolimnion is unknown, but it is likely to be in recently deposited sediment at the sediment surface (Hurley et al. 1994). The mechanism of release is probably a function of biological and chemical factors. For example, biological degradation of particles or changes in chemical conditions (e.g., oxygen concentration, sulfide concentration, partition coefficient) could enhance release.

Quantification of remineralization of mercury or other elements is usually based on a comparison of gross sedimentation with net sedimentation. Gross sedimentation is calculated from sediment traps deployed at appropriate time intervals in a suitable location in the water column. Net sedimentation can be calculated by dating sediment cores or by determining a material or mass balance budget for the lake.

Remineralization was calculated for Little Rock Lake, Wisconsin (Hurley et al. 1994), and Onondaga Lake (PTI 1994) from the difference between gross and net sedimentation as determined by sediment traps and sediment core dating, respectively. Criticism of the technique involves the usual concerns regarding sediment traps (e.g., appropriate length of deployment period, inclusion of resuspended sediment) and the difficulty in determining a precise value for net sedimentation. However, the alternatives are problematic and rely on

unsupported assumptions. Two alternatives considered for this work plan were use of chambers incubated *in situ* or in the laboratory for estimation of net sedimentation and use of differential sediment trap deployment intervals for direct estimation of remineralization. Chambers containing sediment and incubated *in situ* on the lake bottom were attempted in Pallette Lake, Wisconsin. Particles were allowed to settle on the sediment surface within the chamber, the chamber was then capped, and mercury was measured in overlying water over time (Hurley 1996, pers. comm.). Interpretation of the chamber experiments was problematic because it was impossible to distinguish between release of mercury from recently deposited sediment, diffusion of mercury from deeper sediment, and, in the case of methylmercury, methylation of mercury in the water column (Hurley 1996, pers. comm.). Given these concerns, chambers were considered undesirable for estimation of remineralization in Onondaga Lake.

The second alternative considered for this work plan was differential deployment of sediment traps where traps are deployed for both short- (2–5 days) and long- (2 weeks) term intervals. In general, traps deployed for longer periods of time will underestimate sedimentation because of artificial mineralization of sediment in the traps. The difference between mercury masses observed in the long-term traps and mercury masses predicted to be in the long-term traps (based on masses observed in the short-term traps) would be attributed to remineralization. This technique has been used to estimate loss of phosphorus and organic carbon in sediment traps (reviewed by Bloesch and Burns [1980]). Indeed, this method was used in Onondaga Lake to estimate a remineralization rate of 5 percent per day for phosphorus in sediment traps (Wodka et al. 1985). The sediment trap remineralization rate was then used to correct the calculated fluxes of phosphorus. The difficulty with this approach for estimating mercury remineralization is that conditions within the trap would be assumed to mimic those at the sediment water interface. Remineralization within the trap (which, in the sediment trap literature, is considered artificial and is used to correct sedimentation rates calculated from trap data) would then be assumed to reflect that occurring at the sediment water interface. These assumptions make this approach too experimental for this work plan.

To determine remineralization of mercury in Onondaga Lake in 1996, it is proposed to again compare gross and net sedimentation by the use of sediment traps and sediment sampling. For estimating gross sedimentation, the 1992 sediment trap approach will be refined by deploying the sediment traps for 2 weeks rather than at 4-week intervals. Interval times in 1992 were conservatively long to allow enough material to collect for mercury analysis. The 1992 data (PTI 1993a) indicate that material accumulating during a 2-week deployment interval is sufficient for analysis. Bloesch and Burns (1980) recommend a biweekly deployment to avoid remineralization problems within the traps. Traps will therefore be deployed for 2-week intervals throughout summer stratification. One trap will be deployed in each of the two deep basins approximately 1 m above the sediment surface. Resuspension of sediments in the deep basins is considered minimal (Auer et al. 1996) and, therefore, poses no problem for hypolimnetic sediment traps.

To estimate net sedimentation, deep cores collected from the two basins in the lake will be dated and analyzed for total mercury and methylmercury. Then surface sediment concentrations of total mercury and methylmercury will be compared to sediment trap mercury concentrations. The 1992 field data from Onondaga Lake indicated a lower concentration of methylmercury in the surface sediment (i.e., 0.4–15 μg/kg) as compared to the sediment traps (i.e., 3–167 μg/kg) (PTI 1993a). Similar differences have been found in other stratified lakes and have been cited as evidence for settling particles as a source of methylmercury to the hypolimnion and for low rates of methylmercury production in profundal sediments (Verta and Matilainen 1995).

Consideration of Flux Experiments

Additional experimental work to quantify dissolved flux from sediment was deemed to be of little benefit. Results of flux experiments using chambers, as was done in Onondaga Lake in 1992 (described in PTI [1992]), are highly variable, and their efficacy in estimating flux from sediments to the water column in Onondaga Lake is doubtful. Two alternatives for estimating flux were considered: 1) calculating diffusion rate based on pore water profiles and 2) determining flux based on a mass balance analysis of mercury for the lake. Calculating diffusion rate based on pore water profiles (obtained from filtration of sediment core sections) was attempted in 1992. However, pore water analysis was limited to 2-cm sections by the volume of water necessary for total mercury and methylmercury analysis. Consequently, diffusion rates could only be calculated based on the pore water concentration in the top 2 cm and the concentration in overlying water. It is usually preferable to determine a flux rate based on more than two data points. However, the same approach was used for Little Rock Lake and was considered to represent a rough estimate of flux from deeper sediment (Hurley et al. 1994). It does not account for the possibility of elevated mercury concentrations in a surface microlayer made up of recently deposited sediment (Hurley 1996, pers. comm.). Flux from recently deposited sediment is considered here to constitute remineralization as discussed earlier.

Estimates of diffusive flux in Onondaga Lake and Little Rock Lake (Hurley et al. 1994) are presented in Table 1. The Onondaga Lake values were calculated according to Hurley et al. (1994) based on pore water concentrations in the 0–2 cm sediment section and in overlying water concentrations determined from water column samples (PTI 1993a).

TABLE 1. CALCULATED DIFFUSION RATES OF TOTAL MERCURY AND METHYLMERCURY FROM SURFACE SEDIMENT TO OVERLYING WATER

	Calculated Diffusion Rate (ng/m^2-day)	
	Total Mercury	Methylmercury
Onondaga Lake, New York		
S4A	14	12
S73A	16	8
S83A	22	6
S90A	25	4
Little Rock Lake, Wisconsin (Hurley et al.1994)	12	Not determined

The maximum diffusive flux of total mercury calculated for Onondaga Lake was at Station S90A, which is located in the north basin under 20 m of water. This station is representative of the profundal zone and appears to provide a conservatively high estimate of the diffusive flux of total mercury. If the diffusion rates at Station S90A are applied to the entire hypolimnetic sediment surface area (7.57×10^6 m^2) for 4 months (summer stratification), the potential contribution to the hypolimnion from diffusion is 23 g total mercury and 4 g methylmercury. A hypolimnetic mass balance of the lake indicates that the increase in total mercury and methylmercury in the hypolimnion from May to September 1992 was 660 g and 330 g, respectively (Bigham 1995b, pers. comm.). To put these numbers in perspective, the tributary loading of total mercury and methylmercury to the lake during this time period was 4,300 g and 83 g, respectively (Bigham 1995b, pers. comm.). Thus, the calculated diffusion rates are 0.5 percent and 5 percent of tributary loading for total mercury and methylmercury, respectively. Based on these calculations, diffusion of dissolved total mercury and methylmercury from Onondaga Lake sediments is a relatively minor source of mercury to the hypolimnion

Other methods of determining mercury concentrations in pore water from sediments (e.g., peeper, sipper) were considered but are generally problematic because of the potential for contamination of samples (peeper), the difficulty in deployment in deep and anoxic water (peeper and sipper), and clogging in fine-grained sediment (sipper).

An alternative approach to determining flux is to do a well-constrained mass balance on the lake and, by difference, assign a value to or boundaries on flux. This can be done, to some extent, with the 1992 data set. In addition, a sensitivity analysis with the Onondaga Lake Mercury Model can be used to set boundaries on flux estimates. Based on these arguments, further experimentation to quantify flux would have little benefit. Diffusive flux will be estimated based on a mass balance analysis of the 1992 data set once the Onondaga Lake Mercury Model is recalibrated.

SAMPLE LOCATION, FREQUENCY, AND EXPERIMENTAL PROCEDURE

Net Mercury Methylation in the Water Column

Experiments for determining net methylmercury production in Onondaga Lake will be done under the direction of Dr. Cynthia Gilmour of the Academy of Natural Sciences of Philadelphia, Estuarine Research Center (ANSP/ERC). Water column samples for measuring net mercury methylation rates will be collected in June and September of 1996 from the two lake stations used in 1992 (i.e., center of each of the two basins). Samples will be taken from three depths by peristaltic pump with pre-cleaned C-flex and Teflon® tubing directly into 500 mL Teflon® bottles. The three depths will represent the middle of the surface layer, just below the pycnocline, and near the bottom of the hypolimnion. The specific depths will be determined in the field based on vertical profiles of temperature and dissolved oxygen measured with a Hydrolab®.

Mercury methylation will be measured over time, with duplicate measurements made at each time point. Each measurement will be made in a separate 500-mL Teflon® bottle to minimize sample disturbance and contamination. Sample bottles will be spiked with ^{203}Hg to obtain a final added concentration of approximately 2 ng/L. This concentration will represent an approximately 20 percent addition to ambient summer total mercury concentrations in anoxic bottom waters. ^{203}Hg is produced by a custom synthesis to a specific activity between 15 and 30 mCi/mg. At this specific activity, less than 50 nCi ^{203}Hg-mercury per bottle and less than 5 μCi per sampling trip will be used.

Bottles will be held at ambient temperature and will be shaded during sampling and manipulation. After addition of ^{203}Hg to sample bottles, bottles will be incubated at the depth from which water samples were taken. The bottles will be returned to their appropriate depth by placing them in a plastic bag on a rope marked with the correct depth. At each depth sampled, duplicate bottles will be retrieved after approximately 6, 12, 24, and 48 hours. The first sample, taken at the beginning of the experiment, will act as a blank because the bottle will not be returned to the lake for incubation.

Control samples will be established at time zero and retrieved at 24 hours. The control variables are as follows:

- Filtration to establish whether methylation is an abiotic or biotic process
- Addition of molybdate (i.e., a specific inhibitor of sulfate reduction) to establish whether methylation involves sulfate-reducing bacteria.

The filtered control sample will be performed for all depths, while the molybdate control sample will be performed for samples from the pycnocline and hypolimnion only.

Upon retrieval from the lake, the contents of the Teflon® bottles will be acidified, cooled, and prepared for transport. Contents of the bottles will be extracted for ^{203}Hg-methylmercury and analyzed on a gamma counter at the Estuarine Research Center in St. Leonard, Maryland. In addition, blanks consisting of lake water returned to the lab and spiked with ^{203}Hg will be analyzed.

The water column methylmercury production experiment will result in 40 samples (3 depths, 2 replicates, 5 times, plus 2 replicates of 1 control at 3 depths and 2 replicates of 1 control at 2 depths) for each sampling period at each station. Each sample will be analyzed for ^{203}Hg-methylmercury. Sulfide and/or dissolved oxygen concentration will be measured in replicate bottles at each sampling time. Ambient measurements of total and dissolved total mercury and methylmercury, sulfide, total suspended solids, and anions/ cations will also be determined for each site at six depths. Measurements of pH, dissolved oxygen concentration, conductivity, temperature, and depth will be performed in the field with a Hydrolab®.

The sampling plan for methylmercury production is summarized in Table 2.

Remineralization of Mercury in the Hypolimnion

Remineralization of total mercury and methylmercury in Onondaga Lake will be calculated from the difference between gross and net sedimentation. Gross sedimentation will be determined by deployment of one sediment trap approximately 1 m above the sediment surface in each of the two deep basins. These traps will correspond in location to the hypolimnetic traps used in the 1992 field investigation (PTI 1993a). Traps will be deployed and retrieved at 2-week intervals from mid-May through mid-October for a total of 10 retrievals. Trap contents will be analyzed for total mercury, methylmercury, percent solids, and total mass. In addition, samples of water overlying the traps will be analyzed for total suspended solids to permit calculation of settling velocity. These samples will be taken when traps are first deployed and at each subsequent deployment/retrieval.

The sediment trap work will result in 20 sediment samples for total mercury, methylmercury, percent solids, and total mass. Twenty-two water samples will also be analyzed for total suspended solids.

For estimation of net sedimentation, one 60 cm sediment core will be taken once in the summer in each basin. The cores will be sectioned at 1-cm intervals between 0 cm and 10 cm, and at 2.5 cm intervals between 10 and 60 cm. Sections in the top 10 cm will be analyzed for total mercury and methylmercury. The sections between 10 cm and 60 cm will be analyzed for total mercury. The sections between 20 cm and 60 cm will be analyzed for ^{137}Cs-cesium and lead. Analysis of ^{137}Cs-cesium and lead above 20 cm will not yield information useful for dating purposes. ^{137}Cs-cesium will identify 1955 and 1963 (appearance and peak), total mercury will identify 1947 and 1970 (increase and decrease), and lead will identify 1970 (peak) (Klein and Jacobs 1995). The stratigraphic cores will

TABLE 2. SUMMARY OF SAMPLING SPECIFICATIONS FOR THE ONONDAGA LAKE RI/FS MERCURY METHYLATION AND REMINERALIZATION INVESTIGATIONS

	No. of Stations	No. of Samples/ Station	Sampling Period	Total No. of Samples	Analyses
Mercury Methylation Investigation					
Water samples incubated *in situ* at 3 depths, 2 replicates, 5 sample times, plus 10 controls per station, plus 2 laboratory blanks per sampling period	2	40	6/96, 9/96	168	^{203}Hg-Methylmercury Sulfide and/or dissolved oxygen
Ambient measurements	2	6	6/96, 9/96	24	**Field Measurements** pH Dissolved oxygen Conductivity Temperature **Laboratory Analyses** Total mercury (total and dissolved) Methylmercury (total and dissolved) Total suspended solids Anions/cations Sulfide
Mercury Remineralization Investigation					
Sediment from sediment traps	2	1	Biweekly 5/96–10/96	20	Total mercury Methylmercury Percent solids Total mass
Water overlying sediment traps	2	1	Biweekly 5/96–10/96	22	Total suspended solids
Deep sediment cores to 60 cm sectioned into 1.0-cm intervals from 0–10 cm, and 2.5-cm intervals from 10–60 cm (not all intervals will be sampled for all analytes)	2	1	Once/summer	60 20 40 40	Total mercury Methylmercury ^{137}Cesium Lead Percent solids
Short sediment cores to 5 cm, sectioned into five 1-cm intervals	2	2	Once/summer	20	Total mercury Methylmercury Percent solids

produce 60 samples (30 per core) for total mercury analysis, 20 samples (10 per core) for methylmercury analysis, and 40 samples (20 samples per core) for ^{137}Cs-cesium and lead analysis.

In addition, two 3-in. diameter cores will be taken at each site to a depth of 5 cm to give an estimate of spatial variability in sediment mercury concentrations. The short cores will be analyzed for total mercury, methylmercury, and percent solids in 1-cm sections. The short cores will produce 20 samples for total mercury, methylmercury analyses, and percent solids.

The sampling plan for remineralization is summarized in Table 2.

SAMPLE DESIGNATION, DOCUMENTATION, AND HANDLING PROCEDURES

Sample designation, documentation, and handling procedures for the methylation and remineralization study will be consistent with other Onondaga Lake RI/FS studies. Samples will be documented and handled in accordance with the procedures outlined in the Onondaga Lake RI/FS sampling and analysis plan (PTI 1991a) and the quality assurance project plan (PTI 1991b). These protocols ensure maintenance of sample integrity from the time of field collection to the time of laboratory analysis. Documentation will include sample logs, chain-of-custody records, and sample analysis request forms.

Samples from the water column methylation study will be collected in 500 mL Teflon® bottles. Bottles will be acid-cleaned using the same procedure for all Onondaga Lake RI/FS total mercury and methylmercury water analyses. Samples will be held at 4°C following collection and during transport to the analytical laboratory. Analyses will be performed within 56 days.

Special equipment for the methylation study (e.g., 500-mL Teflon® bottles, ^{203}Hg, Hydrolab®, pump) will be supplied by ANSP/ERC under the direction of Dr. Cynthia Gilmour. ANSP/ERC will also provide personnel for the methylation study. ^{203}Hg analysis and water chemistry analysis for the water column methylation study, with the exception of total mercury and methylmercury, will be done by ANSP/ERC personnel. Mercury analyses will be provided by Frontier Geosciences.

Sediment traps will be deployed and retrieved according to the field method described in Appendix B of the *Onondaga Lake RI/FS Mercury and Calcite Mass Balance Investigation Data Report, Volume II* (PTI 1993b). Samples from the sediment traps will be placed in acid-cleaned Teflon® sample bottles as described in the field method. Samples will be held at 4°C following collection and during transport to the analytical laboratory. Analyses will be performed within 28 days.

Sediment core sampling will follow the procedure described in the Onondaga Lake RI/FS sampling and analysis plan (PTI 1991a). Samples will be handled as described above.

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